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Construction and initial testing of a microwave spectrometer

Thomas Willard Duecker
University of the Pacific

CONSTRUCTION AND INITIAL TESTING OF A MICROWAVE SPECTROMETER

A Thesis

Presented to

The Faculty of the Department of Chemistry

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In Partial Fulfillment

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Master of Science

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by

Thomas Willard Duecker

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ACKNOWLEDGMENT

I am indebted to Dr. DeVault and his associates for their original work in 1952 of constructing a microwave spectrometer. Their original notes and data were invaluable.

My sincere appreciation goes to Dr. W.H. Wadman for his patience and constructive criticism during the construction of this spectrometer.

I am also indebted to Professors L.E. Colip and D.G. Dutra of the electrical engineering department of the University of the Pacific's School of Engineering for their assistance.

FOREWORD

This thesis has been written to provide a basic understanding of the spectrometer and to be used as a useful reference for its operation. Discussions of such things as the propagation of microwaves and the mechanics of klystron operations have been avoided.

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I

GENERAL DESCRIPTION OF THE INSTRUMENT

Microwave spectroscopy is in many ways a continuation of the more common forms of spectroscopy known as infra-red, ultra-violet, and optical. As with the other forms of spectroscopy, a compound can be identified by its characteristic frequency or frequencies of absorption. Many compounds which failed to show identifying frequencies under optical, ultra-violet, or infra-red stimulation now are identified by using microwave frequencies. Microwave spectroscopy also has the unique feature of covering a frequency range identical with many of the fundamental frequencies of rotation of molecules. Microwave spectrometers have been built which precisely identify both the frequency of absorption and quantitative amount of energy absorbed by the sample, thus permitting the direct calculation of thermodynamic properties.

The microwave spectrometer described in this thesis is at present a simple system and does not contain many of the possible accessories or refinements which in time could be added.

The basic system consists of a microwave source, an absorption cell, a detector, and a system for presentation of data (See diagram 1).

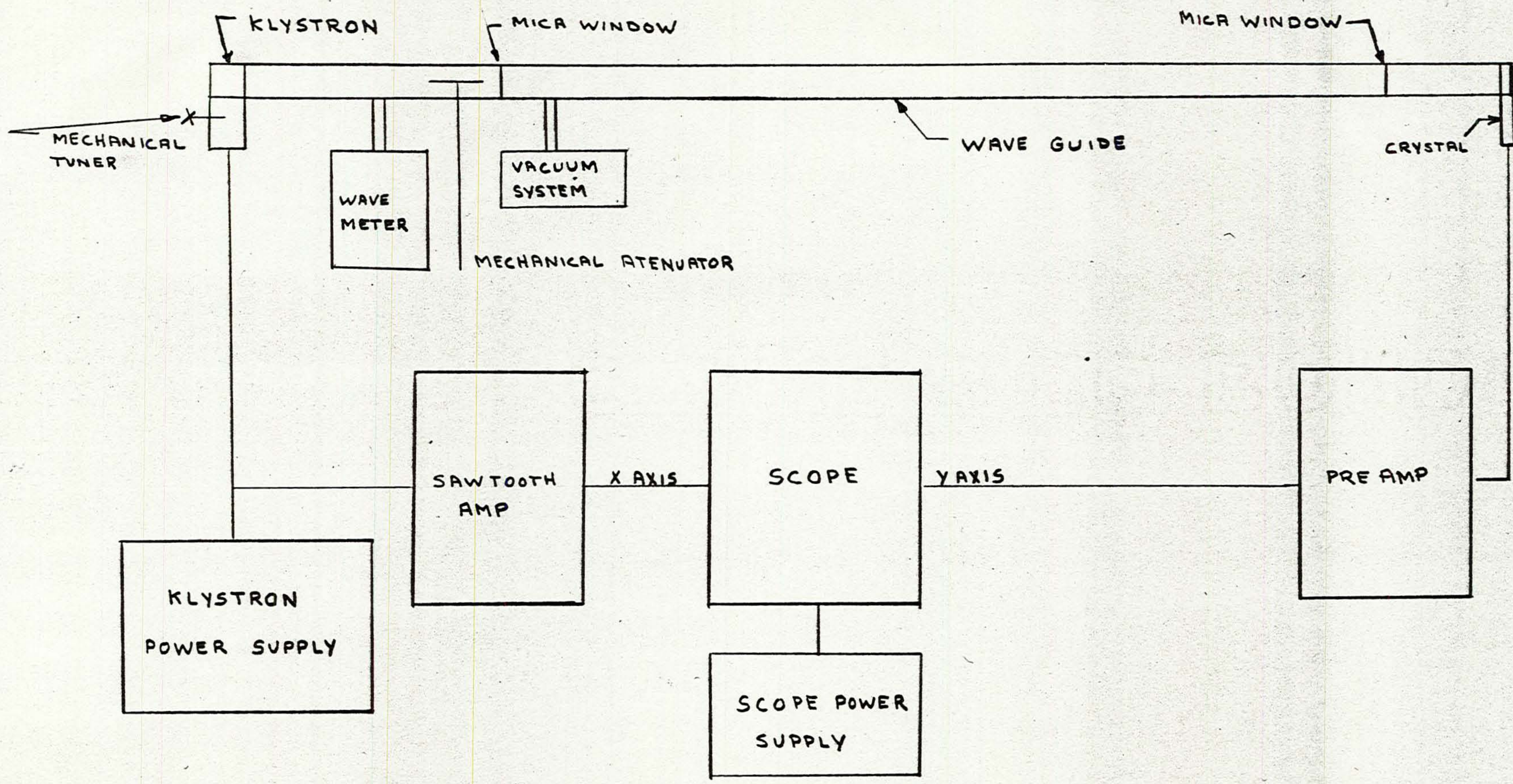


DIAGRAM 1

BLOCK DIAGRAM MICROWAVE SPECTROMETER

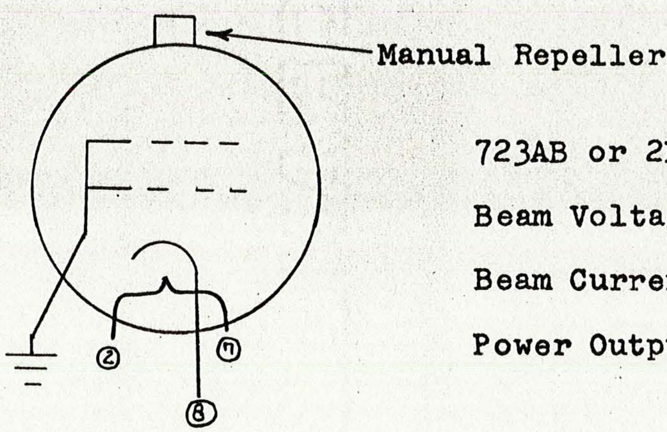
The source was built using a 723A/B or 2K25 reflex klystron tube, generating microwave frequencies between 5000 and 10,000 megacycles per second (See diagram 2). A suitable power supply was built to supply the tube with beam voltage and a variable repeller voltage. The reflex klystron can be tuned by a mechanical adjustment of the struts which changes the physical size of the cavity, or tuned electrically by varying the repeller voltage to one of the various modes of oscillation. The klystron and its related power supply furnish the various microwave frequencies to the wave-guide.

Unlike optical systems where transmission is via air and lenses, or the common radio communications via wire and air, microwaves require the use of a wave-guide. Because the power loss over wire makes its use prohibitive, the wave-guide offers the most efficient means of controlling and directing microwaves.

A frequency meter is placed in the wave-guide to provide a means of identifying the frequency generated by the klystron. The frequency meter is a wave-meter consisting of a cavity tuned by a micrometer adjustment and a meter to indicate variations in power. As the cavity is tuned via the micrometer, a point of resonance is reached where the cavity absorbs the frequency being generated. This resonance causes a drop in power and a corresponding

DIAGRAM 2

KLYSTRON DATA



723AB or 2K25

Beam Voltage 300v.

Beam Current 22ma.

Power Output 28mw.

1. On a new klystron do not turn the manual repeller more than $2\frac{1}{2}$ turns.
2. After loosening, it is permissable to turn the manual repeller a maximum of $3\frac{1}{4}$ turns.
3. As the repeller voltage is varied the beam current gives little maxima when oscillation occurs.
4. Klystrons operate hot, but get hotter if not oscillating.
5. Beam current is normally 28 to 34ma. on peaks.
6. It is possible to tune to the center of the mode by putting a slow sawtooth sweep on the repeller and tuning the repeller bias voltage to a minimum fluctuation of the beam current.

Data from <u>Technique of Microwave Measurement</u>
by C.G. Montgomery

deflection of the meter. By multiplying the micrometer reading by the conversion factors printed on the wave-meter; the output frequency of the klystron is identified.

Next an attenuator is placed in the wave-guide to regulate the output power supplied to the absorption cell. As the attenuator is closed, the power decreases, and correspondingly, as the control is opened, the power is increased. This operation is analagous to the closing or opening of a valve in a pipe line. Thus we now have a microwave signal of known frequency and adjustable intensity.

The absorption cell consists of a metal piece of wave-guide ½" x 1" x 10' long, with mica windows sealed at each end to permit a vacuum system to be attached. In this absorption cell the gas phase of the compound is admitted via the vacuum system; the microwave signal passes through the gas. If the klystron frequency corresponds to a frequency of movement of atoms in the compound, an absorption of power occurs.

After passing through the wave-guide sample cell, the microwaves are terminated in a detector. The detector consists of a 1N21A crystal placed in the microwave field of the wave-guide. This crystal picks up variations in power caused by the gas in the cell and sends minute electrical impulses to a narrow band preamplifier. After passing through the preamplifier the signal is carried by coaxial cable to the presentation device, consisting of an abbreviated Dumont oscilloscope using D.C. amplifiers.

In order that the oscilloscope presentation can be useful, an X and Y axis reference is necessary. This is accomplished by using a sawtooth generator. The sawtooth signal is placed directly on the X axis deflection plates, and also on the repeller of the klystron. The sawtooth is transferred to the klystron, thus producing a frequency modulated signal which passes through the wave-guide, crystal, and preamplifier to the Y axis deflection plates of the oscilloscope. The sawtooth signal serves two functions: the first is the synchronization of the oscilloscope presentation, and the second is that by placing the variable voltage of the sawtooth on the klystron repeller voltage, the output frequency of the klystron is varied back and forth between the limits established by the voltage variation. This eliminates the necessity of minutely tuning the klystron through its frequencies when searching for an absorption frequency.

A sample injection system is required to complete the spectrometer. It is designed to regulate the content and pressure of the absorption cell. This is accomplished by using a vacuum system consisting of a U tube manometer, a drying trap, a Cenco-HiVac pump, and the necessary valves to control vacuum and sample injection. The method of generating the gaseous form of the sample is determined by the requirements of the compound under study.

II

METHOD OF STARTING THE SPECTROMETER

Care must be exercised in operating the spectrometer to prevent accidentally damaging the components. The following procedure should be used to insure safety of operation.

First, turn on the Heathkit Model ps-4 power supply for the preamplifier. After the unit has warmed up for approximately three to five minutes, adjust the B plus voltage to 90 volts and close the switch at the preamplifier.

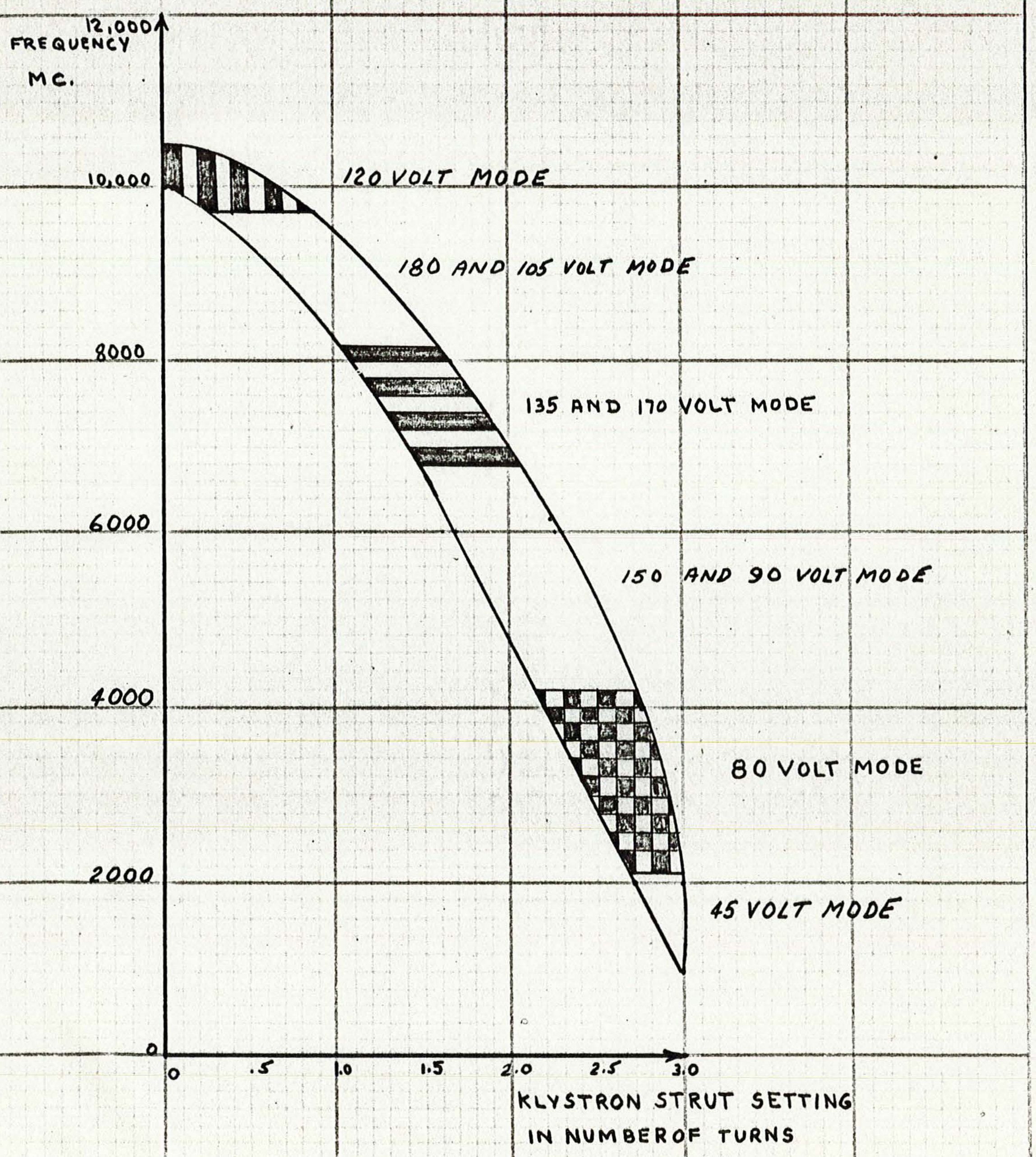
Next, turn the oscilloscope intensity to its full counter-clockwise position. Then close the low voltage switch (right switch) of the oscilloscope power supply. After one minute close the oscilloscope power supply high voltage switch (left switch). Then slowly turn up the oscilloscope intensity until the trace appears.

To operate the klystron, turn the beam voltage and repeller voltage to their full counter-clockwise position. (The regeneration and bias controls are not used and should remain in their full counter-clockwise position at all times.) Turn on the klystron power switch. Adjust the beam voltage to 300 volts. Next turn the klystron sweep off, and while watching the beam amperage meter, increase the repeller voltage until the beam amperage meter gives a peak, indicating the klystron has reached a mode and is oscillating. There are several modes of oscillation which occur at various repeller voltage settings. By use of a particular mode and mechanical setting of the klystron strut control, the various frequencies are produced (See diagram 3 for approximate control settings for a given frequency). The spectrometer is now in full operation.

To turn off the spectrometer the klystron power supply should be turned off first. Then turn off the oscilloscope high voltage. The remaining power switches may then be turned off.

DIAGRAM 3

723 A/B TUBE OUTPUT



III

BASIC METHOD OF OPERATION

A foolproof method of using the spectrometer is impossible to outline. By the nature of the instrument several variables are present for which there is no established point of reference. Only by experiment can one arrive at the proper combination. If a compound has an absorption frequency within the frequency range of the klystron, it should be possible to produce a sharp and clear peak corresponding to this frequency on the oscilloscope tube. This is achieved by maximizing the difference in power between the absorption frequency and the surrounding frequencies. The maximization is the difficult step in operating the spectrometer. For the greatest maximization it is necessary to choose the proper attenuation of the wave-guide, the proper concentration of gas, the proper sweep amplitude, and the proper position of the crystal in the wave-guide.

At the present there is no method of predicting the proper settings of these variables to produce the desired results. One must arrive at the proper combination by experimentation. To add to the problem the choice of oscilloscope settings affects the distinguishability of the oscilloscope trace. Proper choice of X gain, Y gain, oscilloscope attenuation, coarse and fine frequency adjustment, sweep range, and 60 cycle synchronization is necessary to arrive at intelligible results. Before attempting to use the spectrometer one should become familiar with the operation of an oscilloscope, of course.

The following general method can be used to begin searching for an absorption frequency. By the appropriate means a gas suspected of having an absorption frequency within the frequency range of the klystron tube is injected into the wave-guide. Turn on the instrument as previously instructed. To begin searching, close the mechanical repeller struts control (0 setting) and open the wave-guide attenuator to the full counter-clockwise position. Be sure the klystron sweep switch is in the off position. Choose a repeller voltage setting from diagram 3. Adjust the repeller voltage to the chosen value and make a final adjustment by peaking the beam amperage. The klystron is now emitting a microwave frequency. Next turn the oscilloscope attenuation to full counter-clockwise position and turn on the klystron sweep. After a slight pause, slowly increase the klystron sweep amplitude and Y gain until a pattern about one inch high appears on the oscilloscope. This pattern will have several peaks and repeat itself. (The oscilloscope shows two complete cycles.) Now slowly vary the klystron strut control from zero to three turns while watching the scope pattern. Look for an increase on the Y axis of the oscilloscope which will indicate a signal absorption. If no Y gain occurs, choose another repeller voltage setting and again vary the klystron struts. By this means all frequencies of the klystron tube can be covered. Two warnings should be noted. First, the klystron struts should never be opened more than three and one quarter turns, otherwise the vacuum seal is broken and the tube destroyed. Second, in order that one may peak the beam current when setting the repeller voltage, the klystron sweep must be turned off, but one must remember to turn it back on when starting to search another mode.

As the instrument is tuned through the various frequencies and a Y gain appears, the following general procedure should be used. When a peak appears, it should be investigated more closely. This is accomplished by using the oscilloscope controls and klystron sweep amplitude to look at only the immediate area of the peak. That is to say, one should figuratively take that section of the oscilloscope presentation and put it under a magnifying glass. As the sweep amplitude is decreased the klystron emits a narrower range of frequencies. It is therefore desirable to decrease the sweep amplitude and increase the Y gain as much as practicable. If the peak is definitely present it indicates an absorption of a microwave frequency. The problem now is to locate that frequency as closely as possible. Since we are sweeping the klystron it is emitting a range of frequencies. Adjust the klystron struts so that the peak is at a maximum on the left side of the oscilloscope trace. Next turn the klystron sweep off. Adjust the wave-meter sensitivity so that the meter reads between 30 and 50 micro-amps. (See diagram 4). Slowly adjust the micro-meter until the meter needle deflects to the left. Adjust the micrometer so that the needle registers a minimum. Using the sensitivity adjustment, increase the reading to 30 to 50 micro-amps. again. Now readjust the micrometer until the absolute minimum meter reading is obtained. Read the micrometer setting and use the chart to convert to megacycles.

Example:

Micrometer reading 15410
Conversion factor (column Z) .48
Frequency 15410 x .48 7396mc.

One can see that the more pronounced the oscilloscope presentation the more easily an absorption point can be found. As was mentioned earlier the gas concentration and wave-guide attenuation are factors in increasing the absorption intensity and the oscilloscope presentation peak. Any of the several texts on microwave spectrometry which discuss pressure broadening and saturation effects will give further useful information on these factors.

Failure to locate an absorption frequency may be due

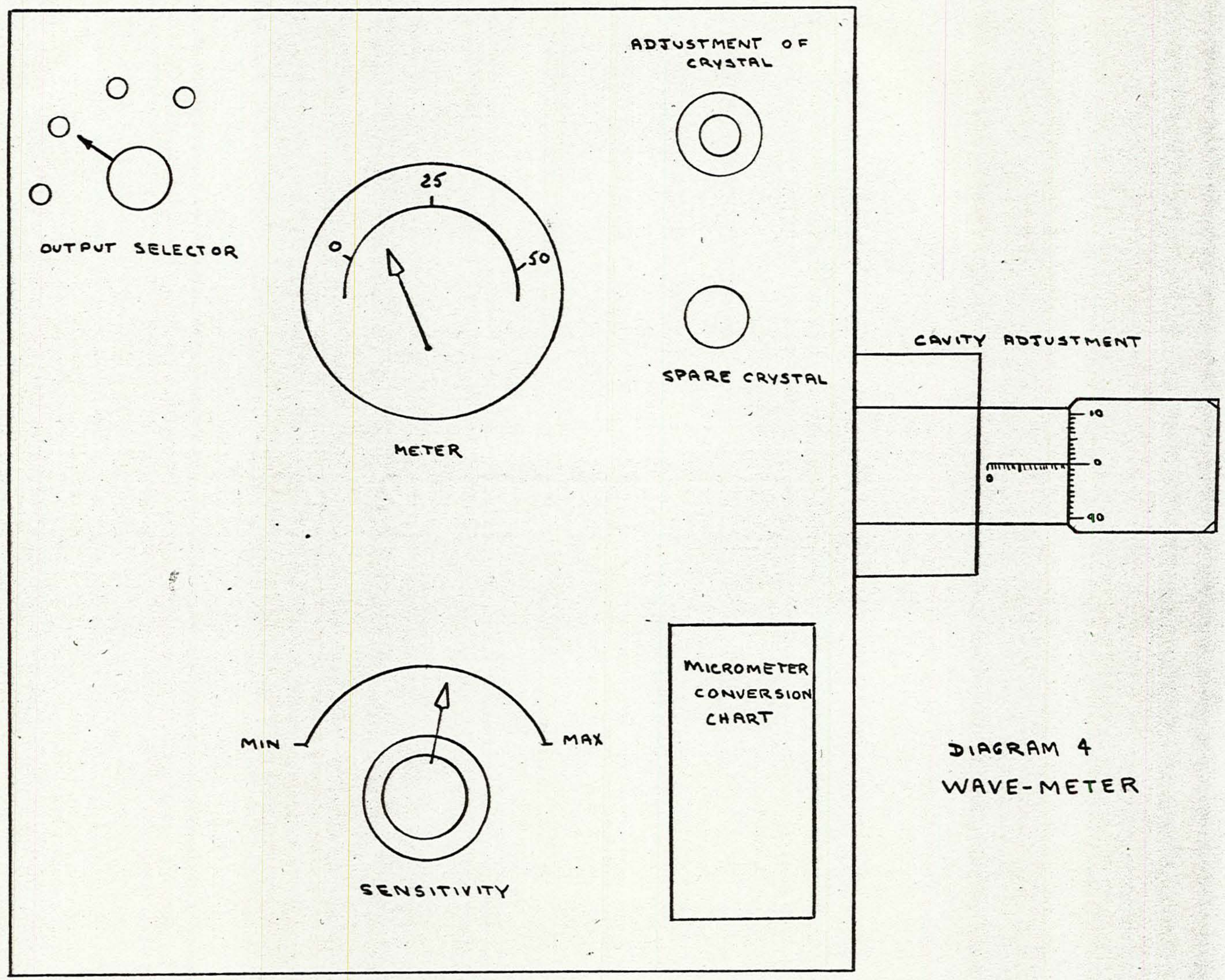


DIAGRAM 4
WAVE-METER

to the wrong combination of variables or the limitations of the instrument.

IV

IDENTIFICATION OF 8885mc. FREQUENCY FOR FORMALDEHYDE

As a means of testing the operation of the spectrometer formaldehyde vapor was chosen for investigation. This compound offers three absorption frequencies within the range of this instrument; these frequencies have been identified and are listed by the National Bureau of Standards in circular 518. The sensitivity of the instrument was found to be insufficient to reproduce the 7363 and 7893 mc. absorption frequencies with any consistency. The 8885mc. absorption frequency was successfully identified.

The following is a description of the test procedure used in identifying the formaldehyde absorption frequency.

A sample injection system was built utilizing a Cenco-HiVac pump, a 250ml. heating mantle, and a 250ml. distilling flask (See diagram 5). A 3/8" hard rubber hose was attached to the wave-guide fitting and connected to a valving center. The valving center consisted of two 12mm. ground glass valves connected to a tee. One valve served as an inlet and the other as an exhaust. Tapped into the tee was a 3mm. stopcock. The stopcock was attached to a mercury manometer, and the remaining leg of the tee was connected to the 3/8" rubber hose from the wave-guide. From the exhaust valve, rubber hose was used to attach a

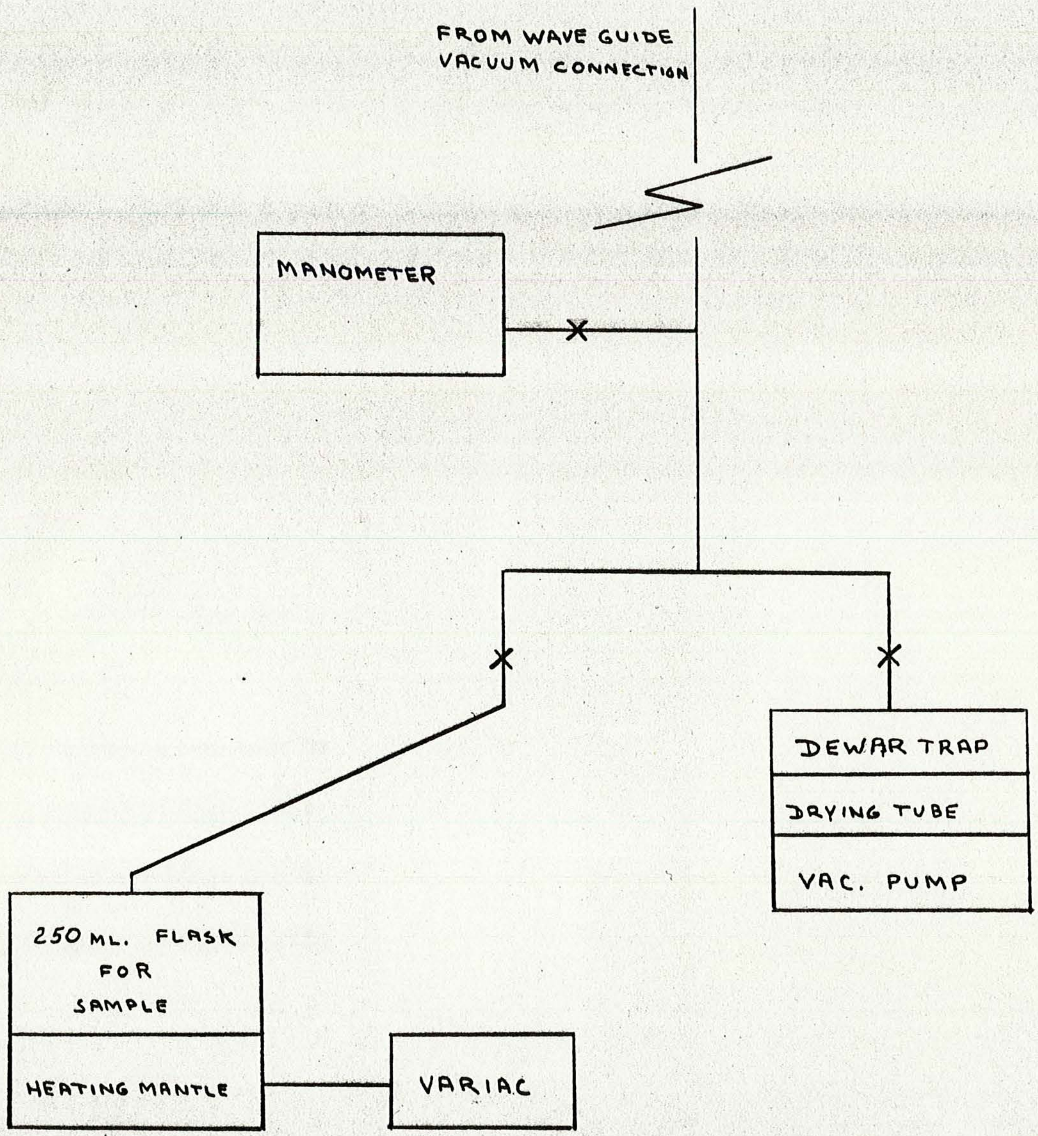


DIAGRAM 5

VACUUM SYSTEM

condensing tube. The condensing tube was placed in a one quart Dewar flask. From the condensing tube a hose was run to the vacuum pump, with a calcium chloride drying tube placed in the line. The drying tube is necessary to protect the vacuum pump oil from contamination and water.

The gas generation system utilized a heating mantle controlled by a Variac and a 250ml. distilling flask. The flask was connected to the inlet valve via a rubber hose.

The design of the injection system permitted the necessary controls to regulate the sample cell. The degree of vacuum was controlled by the exhaust valve with the wave-guide pressure read directly from the manometer. The amount of sample injected was controlled by the inlet valve. Silicon stopcock grease was used on all valves.

To prepare for the sample the cell was pumped down three times. The one quart Dewar was filled with an acetone dry ice mixture at -77^{o}c.. The condensor placed in this solution trapped most of the water vapor in the system. The inlet valve was supplied with dry nitrogen. The cell was pumped down, purged with nitrogen, pumped down again, purged with nitrogen, and pumped down a third time. To generate the formaldehyde vapor, powdered paraformaldehyde was placed in the distilling flask and the flask was stoppered. As the heat vaporised the paraformaldehyde, the inlet valve was opened to permit the filling of the sample cell. After the cell had returned to near atmospheric pressure the inlet valve was tightly closed. By using the vacuum pump and exhaust valve the sample cell pressure and concentration was varied. Next, referring to diagram 3, the 135v. and the 170v. modes were found to cover the required frequency range.

The wave-guide was pumped down to 10^{-2}mm. after sample injection. The attenuation on the wave-guide was opened to its full counter-clockwise position. The oscilloscope attenuation was set at full counter-clockwise position. The instrument was turned on and adjusted to the 170v. mode. A careful tuning of the mechanical struts from zero to three turns did not result in any absorption frequencies being detected. A check of the klystron output for the range of 7000 to 9000mc. by use of the wave-meter showed a low output. The conclusion is that the 170v. mode did not provide enough power to permit detection of the absorption frequencies.

The instrument was then readjusted to the 135v. mode and tuned from zero to three turns of the struts. This resulted in a definite absorption point between .9 and 1.25 turns and a probable point between 1.40 and 1.60 turns of the struts. Attempts were made to locate the 7363 and 7892mc. absorption frequencies. The scope presentation indicated probable peaks between 1.4 and 1.6 strut turns, which compared with the 7892 and 7363mc. frequencies, but results were inconclusive. Attempts were made to vary the gas concentration of the cell, the gas pressure, the wave-guide attenuation, the sweep amplitude, and the Y gain, with no results. An analysis of the variables suggests that the instrument is not sensitive enough to define the 7369 and 7892mc. frequencies. The strength of the absorptions is only 5.1×10^{-7} for the 7363mc. and 1×10^{-6} for the 7892mc. according to the National Bureau of Standards publication circular 518.

Finally, a more detailed investigation of the 8885mc. absorption frequency was conducted. The oscilloscope controls were used to spread the presentation and investigate a smaller section, and a definite peak was found. The klystron sweep amplitude was reduced as far as possible to narrow the sweep range. The Y gain was increased to provide approximately a 2" high peak on the oscilloscope. The X gain was used to spread the presentation. When the klystron strut was tuned between .9 and .125 turns the peak would appear, maximize, and disappear. The strut was turned until the peak was a maximum (See diagram 6). The klystron sweep was turned off, and the wave-meter adjusted to a minimum meter needle deflection.

The absorption point was repeatedly identified by this means. Variations in the sample cell pressure up to atmospheric pressure produced no deviation.

The conclusion is that the 8885mc. absorption

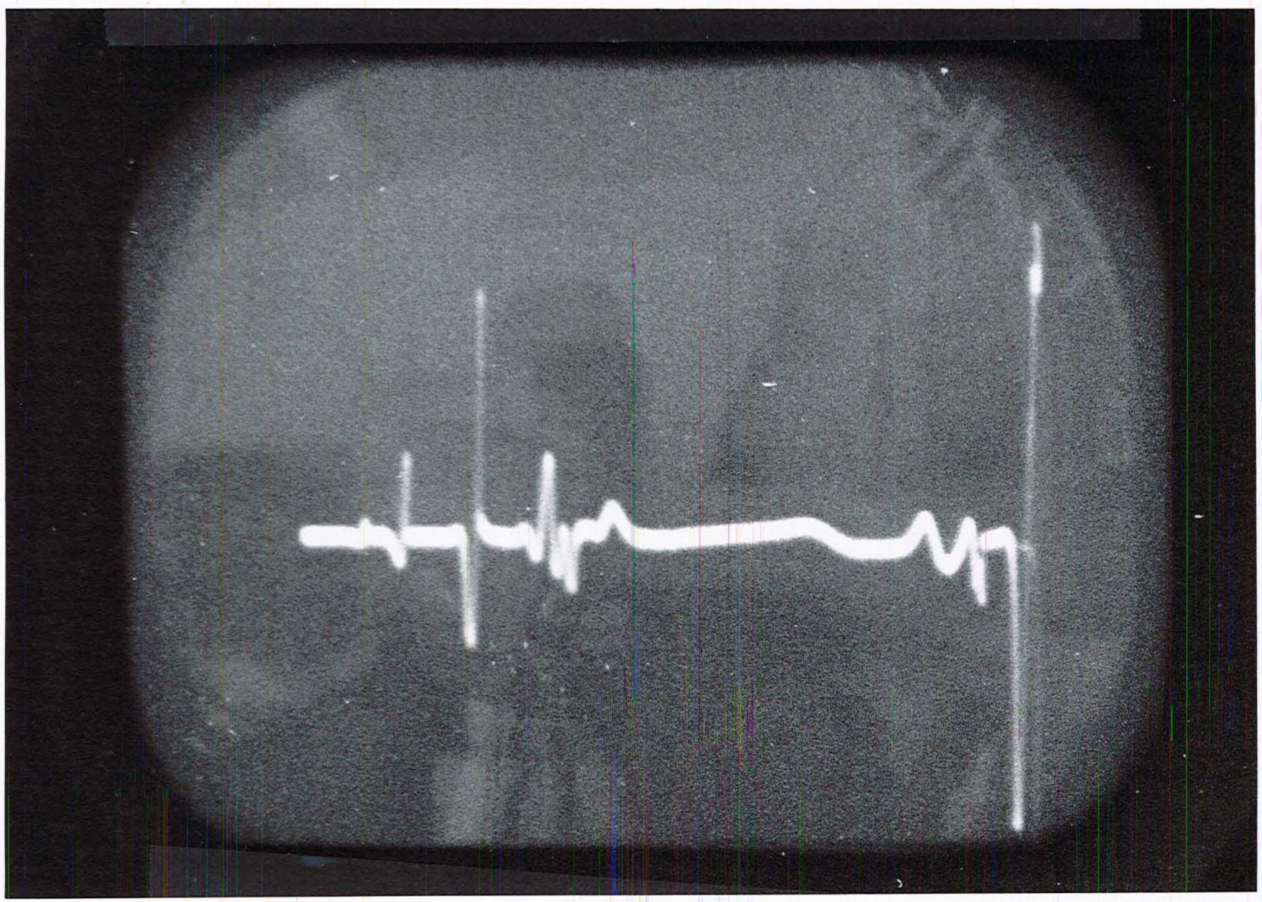


DIAGRAM 6

Photograph of 8885mc. Absorption

frequency of formaldehyde is identifiable by this instrument, and that cell pressure has no identifiable effect on this frequency. The following is a summary of the experimental data (See diagram 7).

DIAGRAM 7

EXPERIMENTAL DATA SUMMARY

Data for 8885mc. frequency

Beam volts--300v.

Beam amperage--31ma.

Repeller volts--135v.

Sample cell pressure--zero to atmospheric

Wave-guide attenuator--full open

Oscilloscope attenuation--full counter-clockwise position

Oscilloscope frequency--coarse

Absorption strength--1×10^{-6} According to the National Bureau of Standards Circular 518.

Approximate Manual Repeller Setting*	Wave-meter	Frequency	N.B.S. Frequency	Absolute Variation
.95	18.00	8910mc.	8885mc.	25mc.
.93	18.11	8964mc.	8885mc.	99mc.
1.10	17.95	8885mc.	8885mc.	0mc.
1.15	17.50	8662mc.	8885mc.	223mc.
1.20	17.70	8762mc.	8885mc.	126mc.
.91	18.15	8984mc.	8885mc.	99mc.
Av. 1.06	17.90	8861mc.	8885mc.	95.3mc.

%Error Frequency $\frac{95.3}{8885} = 1.8\%$ %Error Wave-meter $\frac{.05}{17.95} = .29\%$

* Only approximate due to backlash in tuner

V

SUMMARY

Experimental results show the instrument has an average error of 1.8%. It is believed that this error stems from several sources. The wave-meter conversion factor is an average value for a range of frequencies and accounts for 83% of the error. Human judgment of oscilloscope maximization, non-linearity of crystal response, and amplifier distortion should contribute to the remaining 17% of the error.

In summary this instrument should be useful between 1000 and 10,000mc. It is expected that results should be obtainable within 2% accuracy. The instrument should observe frequencies of absorption down to a minimum intensity of 1×10^{-6} as defined by the National Bureau of Standards Circular 518. No major internal reflection or excessive crystal noises were observed. It is felt that in its present form the spectrometer can be used as an introductory instrument to the field of microwave spectometry and for minor research. It is this author's hope that additional refinements will be made to the instrument to enable its full use as a research tool (See appendix C).

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APPENDIXES

VII

APPENDIX A--Trouble Shooting

Failure of the spectrometer to function correctly is usually caused by one of the following conditions.

A. Connections: Since the system's components are tied together by cabling, an operational failure can be caused by a loose connection. The following points should be checked.

1. Klystron chassis fuse-located front of panel
2. Oscilloscope high, low and ground voltage fuses-located front of oscilloscope power supply panel
3. 12 pronged connector between oscilloscope power supply and oscilloscope chassis-located back of chassis rack
4. Oscilloscope tube high voltage and filament cables-located back of chassis rack
5. A.C. input to klystron power chassis-located back of chassis rack
6. Y axis coaxial input to oscilloscope chassis from preamplifier-located back of oscilloscope chassis
7. A.C. input plug to oscilloscope power supply-located back of chassis rack
8. Klystron power-located back of rack (8 pronged plug)
9. Connecting cable from wave-guide to wave-meter

connection at wave-guide

10. Heathkit B plus and filament connections for preamplifier-both ends of cable-one atop main chassis-second at the preamplifier

11. Coaxial cables-located at the preamplifier

    a. Crystal output at wave-guide

    b. Preamplifier input

    c. Preamplifier output

12. Main A.C. connection to chassis rack and Heathkit

B. Failure of electrical components

Just as with any electronic equipment a competent person should check the following.

1. All vacuum tubes

2. 1N21A crystals (Use caution in handling to prevent damage due to static charge.)

3. Wiring diagrams for component failure.

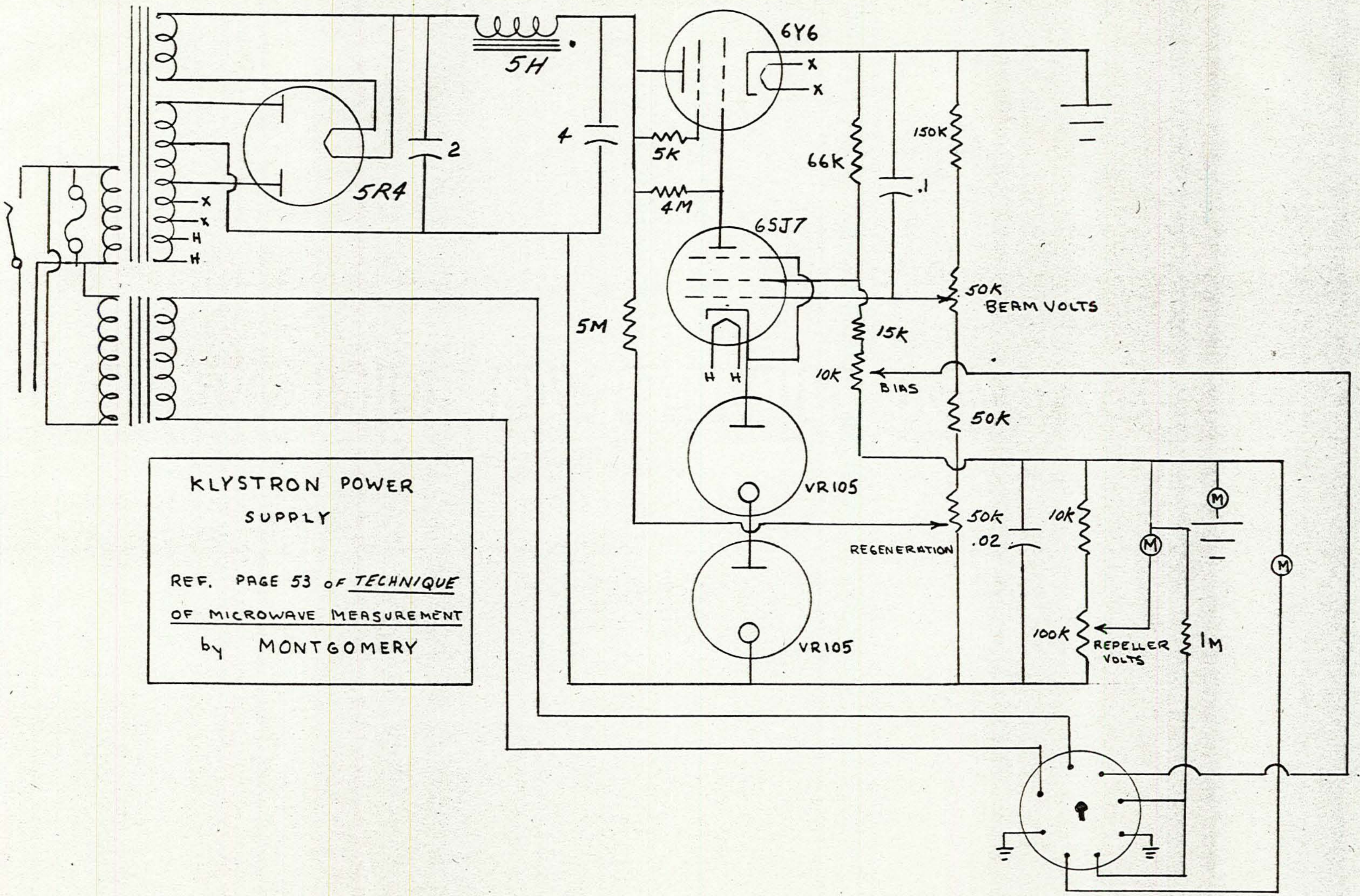
C. Loss of klystron output

The klystron tube is designed to cover only a certain frequency range. Beyond this range the power output to the wave-guide diminishes. For microwave spectrometer use, one can broaden the useful frequency range considerably since power requirements are very low. It must be remembered though at either extreme of a particular mode the power output is dropping rapidly and may be insufficient to be useful. A good indication of the relative output power can be obtained by how far towards maximum the wave-meter sensitivity must be adjusted to achieve a meter deflection of over 50%. In some cases the equipment may be operating correctly, but the klystron output is so low that it is not usable.

VIII

APPENDIX B

Schematic Diagrams

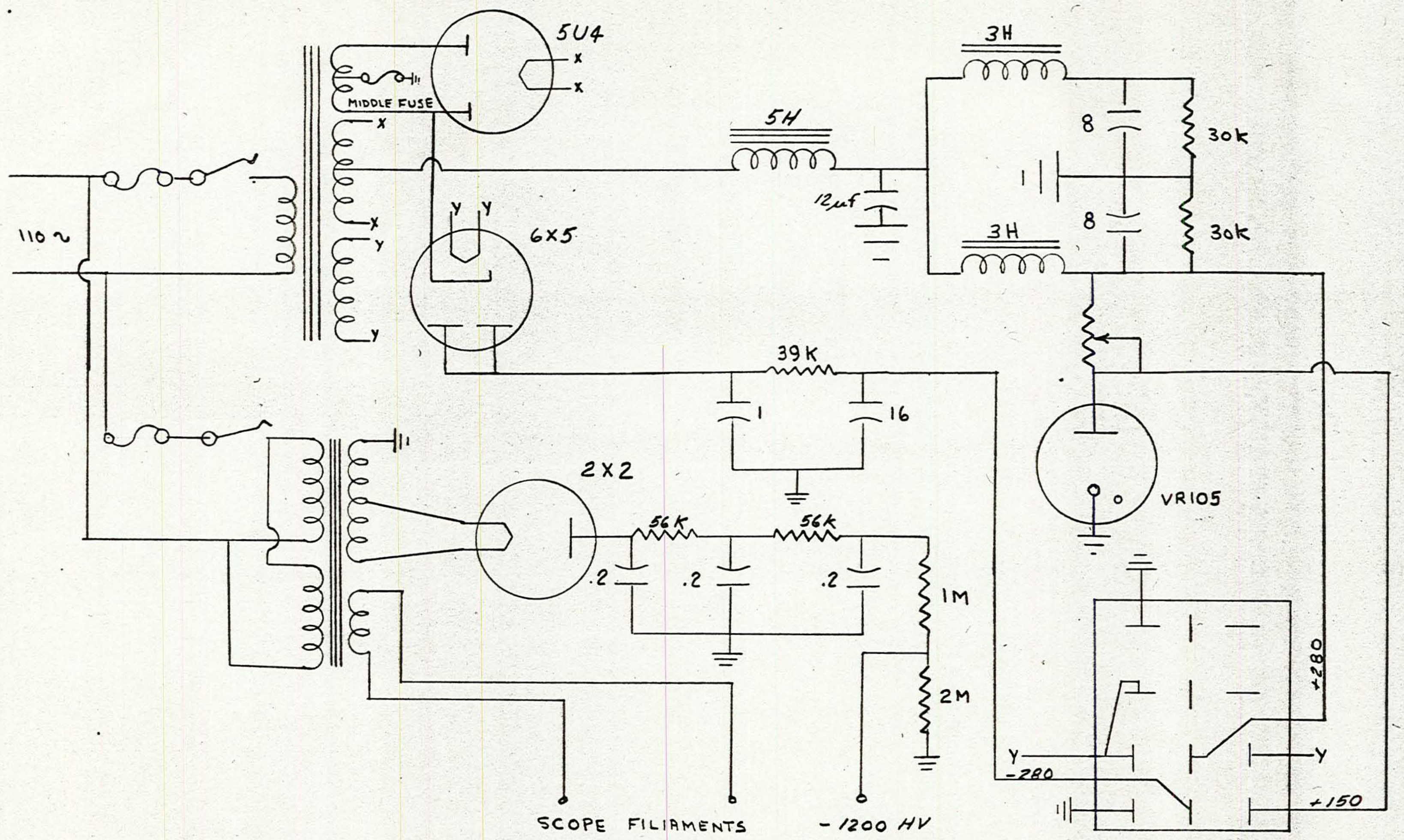


KLYSTRON POWER SUPPLY

REF. PAGE 53 OF <u>TECHNIQUE OF MICROWAVE MEASUREMENT</u>

by MONTGOMERY

DIAGRAM 8



SCOPE POWER SUPPLY

DIAGRAM 9

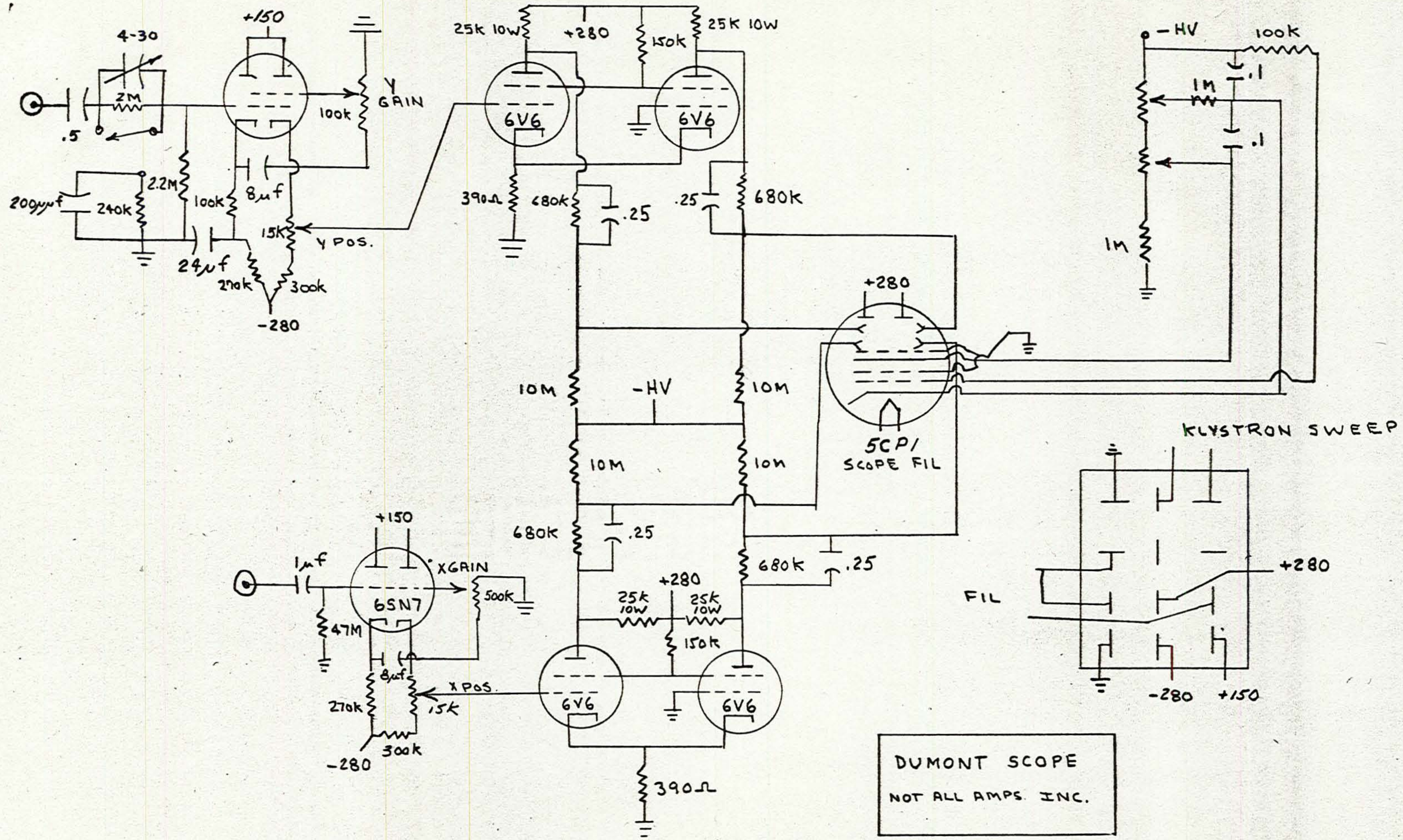
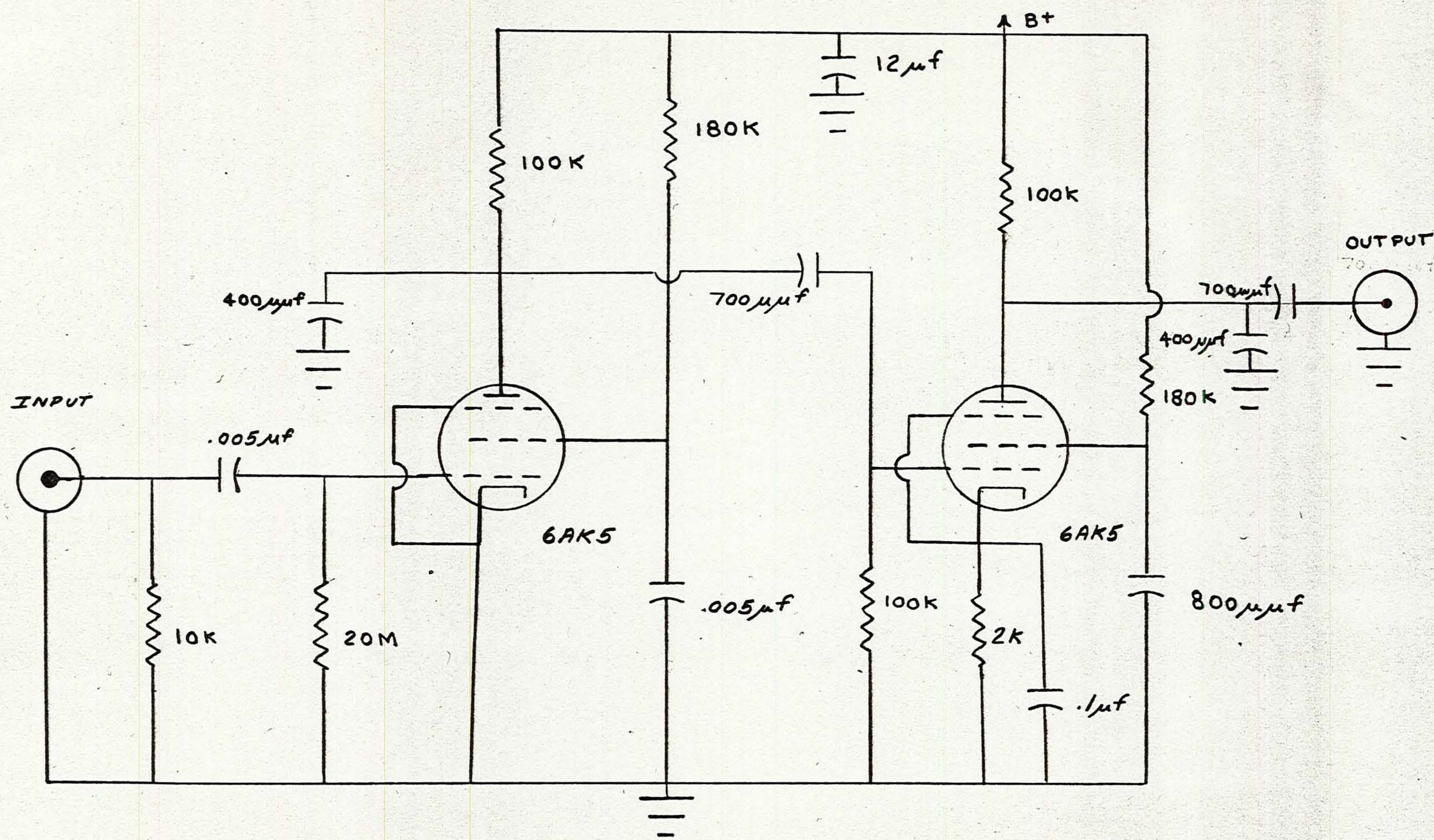
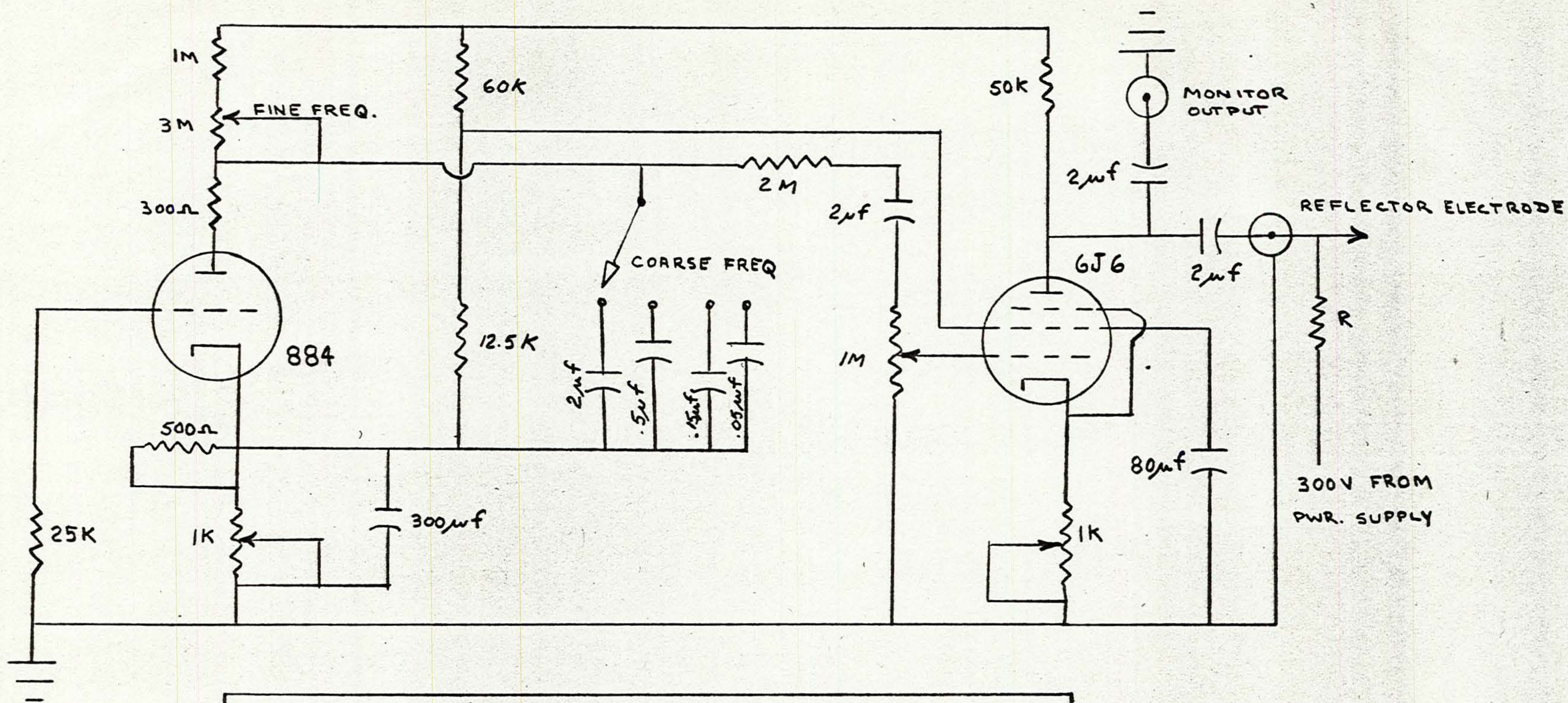

4-30
2M
.5
200μμf
240k
2.2M
100k
+150
24μf
8μf
100k
Y GAIN
15k
Y POS.
270k
300k
-280
25k 10W
+280
150k
25K 10W
6V6
6V6
390Ω
680k
.25
680K
10M
-HV
10M
10M
10M
+280
5CP1
SCOPE FIL
-HV
100k
1M
.1
.1
1M
KLYSTRON SWEEP
+150
1μf
XGAIN
500k
6SN7
47M
8μf
X POS.
270k
15k
300k
-280
680k
.25
+280
25k 10W
25k 10W
150k
6V6
6V6
390Ω
FIL
+280
-280
+150
DUMONT SCOPE
NOT ALL AMPS. INC.

DIAGRAM 10



PRE - AMPLIFIER

DIAGRAM 11



SAWTOOTH GENERATOR

REF. PAGE 125 G.E. RESEARCH LAB REPRINT 1687

by A.H. SHARBAUGH

AUGUST 10, 1949

DIAGRAM 12

APPENDIX C

Future Modification Suggestions

As with any research and construction project the available funds dictate what is practical and feasible. This project was no exception. It is hoped that future modifications of the spectrometer will be made when money and interested personnel are available. The following list offers a beginning point for future modifications.

1. Adaptation of strip chart recorder to supplement the oscilloscope presentation and provide a permanent record of data.
2. A vacuum system capable of much lower pressure than the present Cenco pump arrangement offers.
3. A means of enclosing the wave-guide so that temperature control of the gas phase is possible, thus controlling the degree of excitation.
4. A method of employing a frequency marker so that exact frequency reference is available. This should be based on a pickup of station WWV of the National Bureau of Standards which transmits exact microwave frequencies.
5. A mechanical system which would tune both the repeller voltage and klystron strut settings enabling a complete

scan of the frequency range. (Similar to the automatic tuning of a grating in an optical instrument).

6. The addition of Stark-modulation which would increase the sensitivity of the instrument by 10^2. This would require modification of the wave-guide, construction of a square wave generator and twin tee amplifier for pickup.

The addition of the above components would control some of the variables inherent in the present system and considerably reduce the limits of error.